

## **Analytical Laboratory**

13339 Hagers Ferry Road Huntersville, NC 28078-7929 McGuire Nuclear Complex - MG03A2 Phone: 980-875-5245 Fax: 980-875-4349

## **Order Summary Report**

144000000

Order Number:	J11060066	
Project Name:	WWTS - Biweekly	
Customer Name(s):	Bill Kennedy, Melonie Martin, Wayne Chapman, Tom Johnson	
Customer Address:	3195 Pine Hall Rd	
	Mailcode: Belews Steam Station	
	Belews Creek, NC 28012	
Lab Contact:	Jason C Perkins Phone: 980-875-5348	
Report Authorized By: (Signature)	<b>Date:</b> 6/23/2011	

#### **Program Comments:**

FGD BiMonthly Sampling

#### **Data Flags & Calculations:**

Any analytical tests or individual analytes within a test flagged with an "X" or "1" indicate a deviation from the method quality system or quality control requirement. All results are reported on a dry weight basis unless otherwise noted.

#### Data Package:

This data package includes analytical results that are applicable only to the samples described in this narrative. An estimation of the uncertainty of measurement for the results in the report is available upon request. This report shall not be reproduced, except in full, without the written consent of the Analytical Laboratory. Please contact the Analytical laboratory with any questions. The order of individual sections within this report is as follows:

Job Summary Report, Sample Identification, Technical Validation of Data Package, Analytical Laboratory Certificate of Analysis, Analytical Laboratory QC Reports, Sub-contracted Laboratory Results, Customer Specific Data Sheets, Reports & Documentation, Customer Database Entries, Test Case Narratives, Chain of Custody (COC)

#### Certification:

The Analytical Laboratory holds the following State Certifications: North Carolina (DENR) Certificate #248, South Carolina (DHEC) Laboratory ID # 99005. Contact the Analytical Laboratory for definitive information about the certification status of specific methods.

## Sample ID's & Descriptions:

BELEWS C	08-Jun-11 8:00 AM 08-Jun-11 8:05 AM 08-Jun-11 8:10 AM 08-Jun-11 8:15 AM	W. B. WORKMAN W. B. WORKMAN W. B. WORKMAN W. B. WORKMAN	FGD Purge Eff EQ TANK EFF. BIOREACTOR 1 INF.
BELEWS C	08-Jun-11 8:10 AM	W. B. WORKMAN	
BELEWS 0			BIOREACTOR 1 INF.
	8-Jun-11 8:15 AM	W B WORKMAN	
BELEWS 0		VV. D. VVOININIAIN	BIOREACTOR 2 INF.
	8-Jun-11 8:20 AM	W. B. WORKMAN	BIOREACTOR 2 EFF.
BELEWS 0	2-Jun-11 10:00 AM	S.S.	Trip Blank
BELEWS 0	2-Jun-11 10:00 AM	S.S.	FILTER BLANK
BELEWS 0	8-Jun-11 11:17 AM	ILLEGIBLE	BIOREACTOR 1 INF.
BELEWS 0	8-Jun-11 11:17 AM	ILLEGIBLE	HG BLANK BIOREACTOR 1 INF.
BELEWS 0	8-Jun-11 11:34 AM	ILLEGIBLE	BIOREACTOR 2 INF.
BELEWS 0	8-Jun-11 11:34 AM	ILLEGIBLE	Hg Blk BioReactor 2 Inf
BELEWS 0	8-Jun-11 11:27 AM	ILLEGIBLE	BIOREACTOR 2 EFF.
BELEWS 0	8-Jun-11 11:27 AM	ILLEGIBLE	Hg Blk BioReactor 2 Eff
	BELEWS 0: BELEWS 0: BELEWS 0: BELEWS 0: BELEWS 0: BELEWS 0:	BELEWS       02-Jun-11 10:00 AM         BELEWS       08-Jun-11 11:17 AM         BELEWS       08-Jun-11 11:34 AM         BELEWS       08-Jun-11 11:34 AM         BELEWS       08-Jun-11 11:27 AM	BELEWS       02-Jun-11 10:00 AM       S.S.         BELEWS       08-Jun-11 11:17 AM       ILLEGIBLE         BELEWS       08-Jun-11 11:37 AM       ILLEGIBLE         BELEWS       08-Jun-11 11:34 AM       ILLEGIBLE         BELEWS       08-Jun-11 11:34 AM       ILLEGIBLE         BELEWS       08-Jun-11 11:27 AM       ILLEGIBLE

## **Checklist:**

Reviewed By:

DataBase Administrator

COC and .pdf report are in agreement with sample and analyses (compliance programs and procedure		<b>✓</b> Yes	□ No
All Results are less than the laboratory reporting lir	nits.	Yes	<b>✓</b> No
All laboratory QA/QC requirements are acceptable		<b>✓</b> Yes	□ No
The Vendor Laboratories have been qualified by th Analytical Laboratory	e	Yes	
Report Sections Included:			
✓ Job Summary Report	✓ Sub-contr	acted Laborat	ory Results
✓ Sample Identification	Customer	Specific Data	Sheets, Reports, & Documentation
✓ Technical Validation of Data Package	☐ Customer	Database En	tries
✓ Analytical Laboratory Certificate of Analysis	☐ Test Case	e Narratives	
☐ Analytical Laboratory QC Report	✓ Chain of 0	Custody	
	☐ Electronic	Data Delivera	able (EDD) Sent Separately

Date:

6/23/2011

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## Order # J11060066

Site: FGD Purge Eff Collection Date: 08-Jun-11 8	:00 AM		Sample #:  Matrix:	<b>2011012191</b> OTHER				
Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst	
MERCURY (COLD VAPOR) IN WA	ATER .							
Mercury (Hg)	167	ug/L		5	EPA 245.1	10-Jun-11 12:15	TLINN	
TOTAL RECOVERABLE METALS BY ICP								
Boron (B)	160	mg/L		0.5	EPA 200.7	16-Jun-11 12:30	MHH7131	
DISSOLVED METALS BY ICP-MS								
Selenium (Se)	457	ug/L		10	EPA 200.8	13-Jun-11 13:08	KRICHAR	
TOTAL RECOVERABLE METALS	BY ICP-MS							
Arsenic (As)	248	ug/L		10	EPA 200.8	20-Jun-11 15:06	KRICHAR	
Chromium (Cr)	204	ug/L		10	EPA 200.8	20-Jun-11 15:06	KRICHAR	
Copper (Cu)	194	ug/L		10	EPA 200.8	20-Jun-11 15:06	KRICHAR	
Nickel (Ni)	213	ug/L		10	EPA 200.8	20-Jun-11 15:06	KRICHAR	

10

10

20

EPA 200.8

EPA 200.8

EPA 200.8

Matrix:

20-Jun-11 15:06

20-Jun-11 15:06

20-Jun-11 15:06

OTHER

KRICHAR

KRICHAR

KRICHAR

CLEEMAN

 SELENIUM SPECIATION

 Vendor Parameter
 Complete
 V\_AS&C

3680

< 10

321

Selenium (Se)

**TOTAL DISSOLVED SOLIDS** 

Collection Date: 08-Jun-11 8:05 AM

Silver (Ag)

Zinc (Zn)

ug/L

ug/L

ug/L

TDS 21000 mg/L 10 SM2540C 14-Jun-11 17:00

Site: EQ TANK EFF. Sample #: 2011012192

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst			
MERCURY (COLD VAPOR) IN WAT	<u>rer</u>									
Mercury (Hg)	154	ug/L		2.5	EPA 245.1	10-Jun-11 12:17	TLINN			
TOTAL RECOVERABLE METALS BY ICP										
Boron (B)	150	mg/L		0.5	EPA 200.7	16-Jun-11 12:34	MHH7131			
DISSOLVED METALS BY ICP-MS										
Selenium (Se)	518	ug/L		10	EPA 200.8	13-Jun-11 13:05	KRICHAR			
TOTAL RECOVERABLE METALS E	BY ICP-MS									
Arsenic (As)	191	ug/L		10	EPA 200.8	20-Jun-11 14:56	KRICHAR			
Chromium (Cr)	187	ug/L		10	EPA 200.8	20-Jun-11 14:56	KRICHAR			
Copper (Cu)	160	ug/L		10	EPA 200.8	20-Jun-11 14:56	KRICHAR			
Nickel (Ni)	188	ug/L		10	EPA 200.8	20-Jun-11 14:56	KRICHAR			

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## Order # J11060066

Site: EQ TANK EFF. Sample #: 2011012192

Collection Date: 08-Jun-11 8:05 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst
TOTAL RECOVERABLE METALS E	Y ICP-MS						
Selenium (Se)	3740	ug/L		10	EPA 200.8	20-Jun-11 14:56	KRICHAR
Silver (Ag)	< 10	ug/L		10	EPA 200.8	20-Jun-11 14:56	KRICHAR
Zinc (Zn)	267	ug/L		20	EPA 200.8	20-Jun-11 14:56	KRICHAR

Site: BIOREACTOR 1 INF. Sample #: 2011012193

Collection Date: 08-Jun-11 8:10 AM Matrix: OTHER

Analyte	Result Units Qualifiers RDI		RDL	Method	Analysis Date/Time	Analyst			
TOTAL RECOVERABLE METALS B	BY ICP								
Boron (B)	151	mg/L		0.5	EPA 200.7	16-Jun-11 12:38	MHH7131		
DISSOLVED METALS BY ICP-MS									
Selenium (Se)	533	ug/L		10	EPA 200.8	13-Jun-11 13:01	KRICHAR		
TOTAL RECOVERABLE METALS BY ICP-MS									
Arsenic (As)	< 10	ug/L		10	EPA 200.8	20-Jun-11 14:17	KRICHAR		
Chromium (Cr)	< 10	ug/L		10	EPA 200.8	20-Jun-11 14:17	KRICHAR		
Copper (Cu)	< 10	ug/L		10	EPA 200.8	20-Jun-11 14:17	KRICHAR		
Nickel (Ni)	21.3	ug/L		10	EPA 200.8	20-Jun-11 14:17	KRICHAR		
Selenium (Se)	521	ug/L		10	EPA 200.8	20-Jun-11 14:17	KRICHAR		
Silver (Ag)	< 10	ug/L		10	EPA 200.8	20-Jun-11 14:17	KRICHAR		
Zinc (Zn)	< 20	ug/L		20	EPA 200.8	20-Jun-11 14:17	KRICHAR		
SELENIUM SPECIATION									
Vendor Parameter	Complete	)			V_AS&C				

Site: BIOREACTOR 2 INF. Sample #: 2011012194

Collection Date: 08-Jun-11 8:15 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst
TOTAL RECOVERABLE METALS I	BY ICP						
Boron (B)	149	mg/L		0.5	EPA 200.7	16-Jun-11 12:42	MHH7131
TOTAL RECOVERABLE METALS I	BY ICP-MS						
Arsenic (As)	< 10	ug/L		10	EPA 200.8	20-Jun-11 14:07	KRICHAR
Chromium (Cr)	< 10	ug/L		10	EPA 200.8	20-Jun-11 14:07	KRICHAR
Copper (Cu)	< 10	ug/L		10	EPA 200.8	20-Jun-11 14:07	KRICHAR
Nickel (Ni)	< 10	ug/L		10	EPA 200.8	20-Jun-11 14:07	KRICHAR
Selenium (Se)	30.1	ug/L		10	EPA 200.8	20-Jun-11 14:07	KRICHAR
Silver (Ag)	< 10	ug/L		10	EPA 200.8	20-Jun-11 14:07	KRICHAR
Zinc (Zn)	< 20	ug/L		20	EPA 200.8	20-Jun-11 14:07	KRICHAR

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## Order # J11060066

Site: BIOREACTOR 2 EFF. Sample #: 2011012195

Collection Date: 08-Jun-11 8:20 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	<b>Analysis Date/Time</b>	Analyst	
MERCURY (COLD VAPOR) IN WA	<u>TER</u>							
Mercury (Hg)	< 1	ug/L		1	EPA 245.1	10-Jun-11 12:32	TLINN	
TOTAL RECOVERABLE METALS BY ICP								
Boron (B)	146	mg/L		0.5	EPA 200.7	16-Jun-11 12:46	MHH7131	
TOTAL RECOVERABLE METALS	BY ICP-MS							
Arsenic (As)	< 5	ug/L		5	EPA 200.8	20-Jun-11 13:57	KRICHAR	
Chromium (Cr)	< 5	ug/L		5	EPA 200.8	20-Jun-11 13:57	KRICHAR	
Copper (Cu)	< 5	ug/L		5	EPA 200.8	20-Jun-11 13:57	KRICHAR	
Nickel (Ni)	< 5	ug/L		5	EPA 200.8	20-Jun-11 13:57	KRICHAR	
Selenium (Se)	< 5	ug/L		5	EPA 200.8	20-Jun-11 13:57	KRICHAR	
Silver (Ag)	< 5	ug/L		5	EPA 200.8	20-Jun-11 13:57	KRICHAR	
Zinc (Zn)	< 10	ug/L		10	EPA 200.8	20-Jun-11 13:57	KRICHAR	
SELENIUM SPECIATION								
Vendor Parameter	Complete	е			V_AS&C			

Site: Trip Blank Sample #: 2011012196

Collection Date: 02-Jun-11 10:00 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst			
TOTAL RECOVERABLE METALS B	Y ICP									
Boron (B)	< 0.05	mg/L		0.05	EPA 200.7	16-Jun-11 12:14	MHH7131			
TOTAL RECOVERABLE METALS BY ICP-MS										
Arsenic (As)	< 1	ug/L		1	EPA 200.8	20-Jun-11 13:53	KRICHAR			
Chromium (Cr)	< 1	ug/L		1	EPA 200.8	20-Jun-11 13:53	KRICHAR			
Copper (Cu)	1.78	ug/L		1	EPA 200.8	20-Jun-11 13:53	KRICHAR			
Nickel (Ni)	< 1	ug/L		1	EPA 200.8	20-Jun-11 13:53	KRICHAR			
Selenium (Se)	1.30	ug/L		1	EPA 200.8	20-Jun-11 13:53	KRICHAR			
Silver (Ag)	< 1	ug/L		1	EPA 200.8	20-Jun-11 13:53	KRICHAR			
Zinc (Zn)	2.51	ug/L		2	EPA 200.8	20-Jun-11 13:53	KRICHAR			
SELENIUM SPECIATION										
Vendor Parameter	Complete	9			V_AS&C					

Site: FILTER BLANK Sample #: 2011012197

Collection Date: 02-Jun-11 10:00 AM Matrix: OTHER

Analyte Result Units Qualifiers RDL Method Analysis Date/Time Analyst

**DISSOLVED METALS BY ICP-MS** 

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#### Order # J11060066

Site: FILTER BLANK Sample #: 2011012197 OTHER Collection Date: 02-Jun-11 10:00 AM Matrix: Analyte Result Units Qualifiers **RDL** Method Analysis Date/Time Analyst **DISSOLVED METALS BY ICP-MS** Selenium (Se) ug/L 2 EPA 200.8 **KRICHAR** < 2 13-Jun-11 12:48 Site: BIOREACTOR 1 INF. Sample #: 2011012198 Collection Date: 08-Jun-11 11:17 AM Matrix: OTHER Qualifiers RDL Analyte Result Units Method **Analysis Date/Time Analyst MERCURY 1631** Vendor Parameter V BRAND Complete Site: HG BLANK BIOREACTOR 1 INF. Sample #: 2011012199 Collection Date: 08-Jun-11 11:17 AM Matrix: OTHER Analyte Result Units Qualifiers RDL Method **Analysis Date/Time Analyst MERCURY 1631** Vendor Parameter Complete V\_BRAND Site: BIOREACTOR 2 INF. Sample #: 2011012200 Collection Date: 08-Jun-11 11:34 AM Matrix: OTHER Qualifiers RDL Method Analysis Date/Time **Analyte** Result Units **Analyst** MERCURY 1631 Vendor Parameter V\_BRAND Complete Site: Hg Blk BioReactor 2 Inf 2011012201 Sample #: Collection Date: 08-Jun-11 11:34 AM Matrix: OTHER Analyte Result Units Qualifiers RDL Method **Analysis Date/Time** Analyst **MERCURY 1631** V\_BRAND Vendor Parameter Complete Site: BIOREACTOR 2 EFF. Sample #: 2011012202 Collection Date: 08-Jun-11 11:27 AM Matrix: **OTHER** Qualifiers RDL Method **Analyte** Result Units Analysis Date/Time **Analyst MERCURY 1631** Vendor Parameter V\_BRAND Complete

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## Order # J11060066

Site: Hg Blk BioReactor 2 Eff Sample #: 2011012203

Collection Date: 08-Jun-11 11:27 AM Matrix: OTHER

Analyte Result Units Qualifiers RDL Method Analysis Date/Time Analyst

MERCURY 1631

Vendor Parameter Complete V\_BRAND



June 17, 2011

Duke Energy ATTN: Jay Perkins Scientific Support-Laboratory 13339 Hagers Ferry Road Huntersville NC 28078 jcperkins@duke-energy.com labcustomer@duke-energy.com

RE: Project DUK-HV1101 Client Project: J11060066

Dear Mr. Perkins,

On June 10, 2011, Brooks Rand Labs (BRL) received three (3) flue gas desulfurization (FGD) waste water samples and three (3) corresponding field blanks. During shipping the sample container for *BioReactor 2 EFF* was broken and unsalvageable. The client was contacted and informed BRL to proceed with the analysis of the five remaining samples. Samples were loggedin for total mercury (Hg) analysis and were received, prepared, analyzed, and stored according to BRL SOPs and EPA methodology.

The results were blank-corrected as described in the calculations section of the applicable SOP(s) and may be evaluated using adjusted reporting limits to account for sample aliquot size. Please refer to the *Sample Results* page for sample-specific detection limits and other details. All quality assurance criteria were satisfied, and all data was reported without additional qualification, aside from concentration qualifiers.

BRL, an accredited laboratory, certifies the reported results of all analyses for which BRL is NELAP accredited meet all NELAP requirements. For more details, see the *Report Information* page of the report. Please feel free to contact me if you have any questions regarding this report.

Sincerely,

Tiffany Stilwater Project Manager

tiffany@brooksrand.com



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## Report Information

### **Laboratory Accreditation**

BRL is accredited by the *National Environmental Laboratory Accreditation Program* (NELAP) through the State of Florida Department of Health, Bureau of Laboratories (E87982) and is certified to perform many environmental analyses. BRL is also certified by many other states to perform environmental analyses. For a current list of our accreditations/certifications, please visit our website at <a href="http://www.brooksrand.com/default.asp?contentID=586">http://www.brooksrand.com/default.asp?contentID=586</a>. Results reported relate only to the samples listed in the report.

## **Field Quality Control Samples**

Please be notified that certain EPA methods require the collection of field quality control samples of an appropriate type and frequency; failure to do so is considered a deviation from some methods and for compliance purposes should only be done with the approval of regulatory authorities. Please see the specific EPA methods for details regarding required field quality control samples.

#### **Common Abbreviations**

BLK	method blank	MS	matrix spike
BRL	Brooks Rand Labs	MSD	matrix spike duplicate
BS	laboratory fortified blank	ND	non-detect
CAL	calibration standard	NR	non-reportable
CCV	continuing calibration verification	PS	post preparation spike
COC	chain of custody record	REC	percent recovery
CRM	certified reference material	RPD	relative percent difference
D	dissolved fraction	RSD	relative standard deviation
DUP	duplicate	SCV	secondary calibration verification
ICV	initial calibration verification	SOP	standard operating procedure
MDL	method detection limit	SRM	standard reference material
MRL	method reporting limit	Т	total recoverable fraction

## **Definition of Data Qualifiers**

(Effective 9/23/09)

- B Detected by the instrument, the result is > the MDL but ≤ the MRL. Result is reported and considered an estimate.
- **E** An estimated value due to the presence of interferences. A full explanation is presented in the narrative.
- **H** Holding time and/or preservation requirements not met. Result is estimated.
- **J** Estimated value. A full explanation is presented in the narrative.
- J-M Duplicate precision (RPD) for associated QC sample was not within acceptance criteria. Result is estimated.
- J-N Spike recovery for associated QC sample was not within acceptance criteria. Result is estimated.
- M Duplicate precision (RPD) was not within acceptance criteria. Result is estimated.
- N Spike recovery was not within acceptance criteria. Result is estimated.
- **R** Rejected, unusable value. A full explanation is presented in the narrative.
- U Result is ≤ the MDL or client requested reporting limit (CRRL). Result reported as the MDL or CRRL.
- X Result is not BLK-corrected and is within 10x the absolute value of the highest detectable BLK in the batch. Result is estimated.

These qualifiers are based on those previously utilized by Brooks Rand, Ltd., those found in the EPA <u>SOW ILM03.0</u>, Exhibit B, Section III, pg. B-18, and the <u>USEPA Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analyses; USEPA; July 2002. These supersede all previous qualifiers ever employed by BRL.</u>



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# Sample Information

Sample	Lab ID	Report Matrix	Type	Sampled	Received
BioReactor 1 Inf	1124031-01	FGD Wastewater	Sample	06/08/2011	06/10/2011
Hg Blk BioReactor 1 Inf	1124031-02	DIW	Field Blank	06/08/2011	06/10/2011
BioReactor 2 Inf	1124031-03	FGD Wastewater	QC Sample	06/08/2011	06/10/2011
Hg Blk BioReactor 2 Inf	1124031-04	DIW	Field Blank	06/08/2011	06/10/2011
Hg Blk BioReactor 2 Eff	1124031-05	DIW	Field Blank	06/08/2011	06/10/2011

# **Batch Summary**

Analyte	Lab Matrix	Method	Prepared	Analyzed	Batch	Sequence
Hg	Water	EPA 1631	06/13/2011	06/14/2011	B110838	1100399



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# Sample Results

Sample	Analyte	Report Matrix	Fraction	Result	Qualifier	MDL	MRL	Unit	Batch	Sequence
<b>BioReactor 1 In</b> 1124031-01	n <b>f</b> Hg	FGD Wastewater	Т	120		1.52	4.04	ng/L	B110838	1100399
<b>BioReactor 2 In</b> 1124031-03	n <b>f</b> Hg	FGD Wastewater	Т	197		3.03	8.08	ng/L	B110838	1100399
<b>Hg Blk BioRead</b> 1124031-02	ctor 1 Inf Hg	DIW	Т	0.15	U	0.15	0.40	ng/L	B110838	1100399
<b>Hg Blk BioRead</b> 1124031-05	c <b>tor 2 Eff</b> Hg	DIW	Т	0.15	U	0.15	0.40	ng/L	B110838	1100399
<b>Hg Blk BioRead</b> 1124031-04	ctor 2 Inf Hg	DIW	Т	0.15	U	0.15	0.40	ng/L	B110838	1100399



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# Accuracy & Precision Summary

Batch: B110838 Lab Matrix: Water Method: EPA 1631

Sample	Analyte	Native	Spike	Result	Units	<b>REC &amp; Limits</b>	RPD & Limits
B110838-SRM1	Certified Reference Mater	ial (112501	5, NIST 1641d	d 1000x dilut	ion)		
	Hg	•	15.68	15.55	ng/L	99% 85-115	
B110838-MS1	Matrix Spike (1124031-03)	)					
	Hg	197.2	1010	1208	ng/L	100% 71-125	
B110838-MSD1	Matrix Spike Duplicate (1	124031-03)					
	Hg	197.2	1010	1257	ng/L	105% 71-125	4% 24

# Method Blanks & Reporting Limits

Batch: B110838 Matrix: Water Method: EPA 1631 Analyte: Hg

Sample	Result	Units
B110838-BLK1	0.07	ng/L
B110838-BLK2	0.04	ng/L
B110838-BLK3	0.10	ng/L
B110838-BLK4	0.05	ng/L

 Average: 0.07
 Standard Deviation: 0.03
 MDL: 0.15

 Limit: 0.50
 Limit: 0.10
 MRL: 0.40



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## **Instrument Calibration**

Sequence: 1100399 Total Mercury Speciation by CVAFS

Method: EPA 1631

**Instrument:** THG-10 **Date:** 06/14/2011

Analyte: Hg

<b>Lab ID</b> 1100399-IBL1	True Value	Result 10.72	<b>Units</b> pg of Hg	REC	& Limits
1100399-IBL2		8.78	pg of Hg		
1100399-IBL3		10.52	pg of Hg		
1100399-IBL4		10.17	pg of Hg		
1100399-CAL1	25.00	26.14	pg of Hg	105%	
1100399-CAL2	100.0	95.39	pg of Hg	95%	
1100399-CAL3	500.0	501.9	pg of Hg	100%	
1100399-CAL4	2500	2542	pg of Hg	102%	
1100399-CAL5	10000	9849	pg of Hg	98%	
1100399-ICV1	1568	1555	pg of Hg	99%	85-115
1100399-CCB1		14.3	pg of Hg		
1100399-CCV1	500.0	486.3	pg of Hg	97%	77-123
1100399-CCV2	500.0	487.4	pg of Hg	97%	77-123

**Bottle Glass** 



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# Sample Containers

Lab ID: 1124031-01 Report Matrix: FGD Wastewater Collected: 06/08/2011 Sample: BioReactor 1 Inf Received: 06/10/2011 Sample Type: Sample Des Container Size Lot **Preservation** P-Lot Ship. Cont. 500 mL **Bottle Glass** No Lot# None N/A Cardboard Box Lab ID: 1124031-02 Collected: 06/08/2011 Report Matrix: DIW Sample: Hg Blk BioReactor 1 Inf Sample Type: Field Blank Received: 06/10/2011 Des Container Size Lot **Preservation** P-Lot Ship. Cont. **Bottle Glass** 500 mL No Lot# None N/A Cardboard Box Lab ID: 1124031-03 Report Matrix: FGD Wastewater Collected: 06/08/2011 Sample: BioReactor 2 Inf Sample Type: QC Sample Received: 06/10/2011 Des Container Size **Preservation** P-Lot Ship. Cont. Lot pН **Bottle Glass** 500 mL No Lot# None N/A Cardboard Box Lab ID: 1124031-04 Report Matrix: DIW Collected: 06/08/2011 Sample: Hg Blk BioReactor 2 Inf Received: 06/10/2011 Sample Type: Field Blank Container Size Lot **Preservation** P-Lot pН Ship. Cont. **Bottle Glass** 500 mL No Lot# None N/A Cardboard Box Lab ID: 1124031-05 Collected: 06/08/2011 Report Matrix: DIW Sample: Hg Blk BioReactor 2 Eff Sample Type: Field Blank Received: 06/10/2011 Preservation P-Lot Des Container Size Lot На Ship. Cont.

None

N/A

Cardboard

Box

500 mL

No Lot#



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# **Shipping Containers**

**Cardboard Box** 

**Received:** June 10, 2011 8:45

**Tracking No:** 4726 7966 1248 via FedEx

Coolant Type: None Temperature: ambient

Description: Cardboard Box Damaged in transit? No Returned to client? No Custody seals present? No Custody seals intact? No COC present? Yes

A D.L		Duke Energy Ana	lytical Laboratory			Analytical L						240 Pa	-		
Duk Enei		Mail Code MGO3/ 13339 Hage	ers Ferry Rd	ORDER#	0000	Sample Clas	OTH	ER	Samples Originati		NC SC			⁴Page 2 STRIBL	
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18804 Northcreek Parkway Bothell, WA, 98011 Tel: (425) 483-3300 Fax: (425) 483-9818 www.appliedspeciation.com

June 16, 2011

Jay Perkins Duke Energy Analytical Laboratory Mail Code MGO3A2 (Building 7405) 13339 Hagers Ferry Rd. Huntersville, NC 28078 (704) 875-5245

Project: Belews - FGD (WWTS Bi-Monthly Sampling) (LIMS # J11060066)

Dear Mr. Perkins,

Attached is the report associated with four (4) aqueous samples submitted for selenium speciation analysis on June 9, 2011. The samples were received in a sealed cooler at 9.4°C on June 10, 2011. Selenium speciation analysis was performed via ion chromatography inductively coupled plasma dynamic reaction cell mass spectrometry (IC-ICP-DRC-MS). Any issues associated with the analysis are addressed in the following report.

If you have any questions, please feel free to contact me at your convenience.

Sincerely,

Ben Wozniak Project Manager

Applied Speciation and Consulting, LLC

Ben Wozniek

## Applied Speciation and Consulting, LLC

Report prepared for:

Jay Perkins
Duke Energy Analytical Laboratory
Mail Code MGO3A2 (Building 7405)
13339 Hagers Ferry Rd.
Huntersville, NC 28078

Project: Belews - FGD (WWTS Bi-Monthly Sampling) (LIMS # J11060066)

June 16, 2011

## 1. Sample Reception

Four (4) aqueous samples in 125mL HDPE bottles (provided by Applied Speciation and Consulting) were submitted for selenium speciation analysis on June 9, 2011. The samples were received on June 10, 2011 in a sealed container at 9.4°C.

Applied Speciation and Consulting strongly recommends that samples submitted for selenium speciation analysis remain at a temperature of  $\leq 6^{\circ}$ C to maintain sample integrity prior to analysis.

The samples were received in a laminar flow clean hood void of trace metals contamination and ultra-violet radiation. Upon reception, the samples were designated discrete sample identifiers. An aliquot of each sample was filtered (0.45µm) and these filtrates were stored in a secure, monitored cryofreezer (maintained at a temperature of -80°C) until selenium speciation analysis could be performed via ion chromatography inductively coupled plasma dynamic reaction cell mass spectrometry (IC-ICP-DRC-MS).

## 2. Sample Preparation

All sample preparation is performed in laminar flow clean hoods known to be free from trace metals contamination. All applied water for dilutions and sample preservatives are monitored for contamination to account for any biases associated with the sample results.

<u>Selenium Speciation Analysis by IC-ICP-DRC-MS</u> Prior to analysis, an aliquot of each sample was filtered with a syringe filter (0.45µm) and injected directly into a sealed autosampler vial. No further sample preparation was performed as any chemical alteration of the samples may shift the equilibrium of the system resulting in changes in speciation ratios.

## 3. Sample Analysis

All sample analysis is precluded by a minimum of a five-point calibration curve spanning the entire concentration range of interest. Calibration curves are performed at the beginning of each analytical day. All calibration curves, associated with each species of interest, are standardized by linear regression resulting in a response factor. All sample results are **instrument blank corrected** to account for any operational biases associated with the analytical platform.

Prior to sample analysis, all calibration curves are verified using second source standards which are identified as initial calibration verification standards (ICV).

Ongoing instrument performance is identified by the analysis of continuing calibration verification standards (CCV) and continuing calibration blanks (CCB) at a minimal interval of every ten analytical runs.

<u>Selenium Speciation Analysis by IC-ICP-DRC-MS</u> All samples for selenium speciation analysis were analyzed by ion chromatography inductively coupled plasma dynamic reaction cell mass spectrometry (IC-ICP-DRC-MS) on June 11, 2011. An aliquot of each sample is injected onto an anion exchange column and mobilized by a basic (pH > 7) gradient. The eluting selenium species are then introduced into a radio frequency (RF) plasma where energy-transfer processes cause desolvation, atomization, and ionization. The ions are extracted from the plasma through a differentially-pumped vacuum interface and travel through a pressurized chamber (DRC) containing a specific reactive gas which preferentially reacts with interfering ions of the same target mass to charge ratios (m/z). A solid-state detector detects ions transmitted through the mass analyzer and the resulting current is processed by a data handling system.

Retention times for each eluting species are compared to known standards for species identification.

## 4. Analytical Issues

The overall analyses went very well and no analytical issues were encountered. All sample results have been corrected in accordance with the continuing calibration verification recoveries to account for perceived instrument drift. All quality control parameters associated with these samples were within acceptance limits.

The estimated method detection limits (eMDLs) for selenite, selenate, and selenocyanate are generated from replicate analyses of the lowest standard in the calibration curve. Not all selenium species are present in preparation blanks; therefore, eMDL calculations based on preparation blanks are artificially biased low.

The eMDL for methylseleninic acid and selenomethionine is calculated from the average eMDL of selenite, selenate, and selenocyanate. The calibration does not contain

methylseleninic acid or selenomethionine due to impurities in these standards which would bias the results for other selenium species.

If you have any questions or concerns regarding this report, please feel free to contact me.

Sincerely,

Ben Wozniak

Project Manager

Ben Wozniek

Applied Speciation and Consulting, LLC

# Selenium Speciation Results for Duke Energy Project Name: Belews - FGD (WWTS Bi-Monthly Sampling) Contact: Jay Perkins LIMS #J11060066

Date: June 16, 2011 Report Generated by: Ben Wozniak Applied Speciation and Consulting, LLC

## Sample Results

						Unknown Se
Sample ID	Se(IV)	Se(VI)	SeCN	MeSe(IV)	SeMe	Species (n)
FGD Purge Eff	159	299	6.0	ND (<4.6)	ND (<4.6)	0 (0)
BioReactor 1 Inf	40.5	476	4.1	1.3	ND (<1.2)	2.6 (1)
BioReactor 2 Eff	2.4	ND (<0.74)	ND (<1.2)	ND (<1.2)	ND (<1.2)	0 (0)
Metals Trip Blk	ND (<0.30)	ND (<0.15)	ND (<0.24)	ND (<0.23)	ND (<0.23)	0 (0)

All results reflect the applied dilution and are reported in µg/L

ND = Not detected at the applied dilution

SeCN = Selenocyanate

MeSe(IV) = Methylseleninic acid

SeMe = Selenomethionine

Unknown Se Species = Total concentration of all unknown Se species observed by IC-ICP-MS n = number of unknown Se species observed

# Selenium Speciation Results for Duke Energy Project Name: Belews - FGD (WWTS Bi-Monthly Sampling) Contact: Jay Perkins LIMS #J11060066

Date: June 16, 2011 Report Generated by: Ben Wozniak Applied Speciation and Consulting, LLC

## **Quality Control Summary - Preparation Blank Summary**

Analyte (µg/L)	PBW1	PBW2	PBW3	PBW4	Mean	StdDev	eMDL*	eMDL 10x	eMDL 50x	eMDL 200x
Se(IV)	0.000	0.000	0.000	0.000	0.000	0.000	0.030	0.30	1.5	6.0
Se(VI)	0.000	0.000	0.000	0.000	0.000	0.000	0.015	0.15	0.74	3.0
SeCN	0.000	0.000	0.000	0.000	0.000	0.000	0.024	0.24	1.2	4.9
MeSe(IV)	0.000	0.000	0.000	0.000	0.000	0.000	0.023	0.23	1.2	4.6
SeMe	0.000	0.000	0.000	0.000	0.000	0.000	0.023	0.23	1.2	4.6

eMDL = Estimated Method Detection Limit

## **Quality Control Summary - Certified Reference Materials**

Analyte (µg/L)	CRM	True Value	Result	Recovery
Se(IV)	LCS	9.57	9.43	98.6
Se(VI)	LCS	9.48	9.16	96.7
SeCN	LCS	8.92	8.77	98.3
MeSe(IV)	LCS	6.47	6.36	98.4
SeMe	LCS	9.32	9.12	97.9

<sup>\*</sup>Please see narrative regarding eMDL calculations

# Selenium Speciation Results for Duke Energy Project Name: Belews - FGD (WWTS Bi-Monthly Sampling) Contact: Jay Perkins LIMS #J11060066

Date: June 16, 2011
Report Generated by: Ben Wozniak
Applied Speciation and Consulting, LLC

## **Quality Control Summary - Matrix Duplicates**

Analyte (µg/L)	Sample ID	Rep 1	Rep 2	Mean	RPD
Se(IV)	BioReactor 2 Eff	2.4	2.5	2.4	3.0
Se(VI)	BioReactor 2 Eff	ND (<0.74)	ND (<0.74)	NC	NC
SeCN	BioReactor 2 Eff	ND (<1.2)	ND (<1.2)	NC	NC
MeSe(IV)	BioReactor 2 Eff	ND (<1.2)	ND (<1.2)	NC	NC
SeMe	BioReactor 2 Eff	ND (<1.2)	ND (<1.2)	NC	NC

ND = Not detected at the applied dilution

NC = Value was not calculated due to one or more concentrations below the eMDL

## **Quality Control Summary - Matrix Spike/ Matrix Spike Duplicate**

Analyte (µg/L)	Sample ID	Spike Conc	MS Result	Recovery	Spike Conc	MSD Result	Recovery	RPD
Se(IV)	BioReactor 2 Eff	278.0	261.9	93.3	278.0	268.6	95.8	2.5
Se(VI)	BioReactor 2 Eff	252.3	253.8	100.6	252.3	253.7	100.6	0.0
SeCN	BioReactor 2 Eff	228.8	224.2	98.0	228.8	224.6	98.2	0.2

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